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## PUBLICATIONS AND PATENTS

July-December 1975



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## REQUEST FOR INFORMATION

Results of research investigations at the Northern Regional Research Laboratory are published regularly in the technical literature, and public-service patents are secured to cover patentable inventions and discoveries (see page 56). As a convenient guide to our publications and patents, a list with abstracts is published semiannually. These abstracts describe the current research and indicate the progress achieved. Further information on any of the developments, as well as earlier technical papers, may be obtained by writing us.

In conformance with the policy of the U.S. Department of Agriculture, Northern Laboratory publications are available to scientists and other specialists, librarians, representatives of the press, and others interested.

Reference to commercial equipment or proprietary products is made as part of the exact experimental conditions. Naming a company or product does not imply approval or

recommendation by the U.S. Department of Agriculture over others not mentioned.

Requests for specific reprints should be by number and addressed to the Northern Regional Research Laboratory. Those titles marked with an asterisk [\*] are not available at the Northern Laboratory for distribution.

Most of the publications are in journals that are available in libraries. Photographic copies of most journal articles on research at this Laboratory can be purchased from the National Agricultural Library of the U.S. Department of Agriculture, Beltsville, Maryland 20705.

No publications will be sent regularly in response to foreign requests unless exchange arrangements have been made with the Director of the National Agricultural Library.

Copies of previous lists of publications and patents are available upon request.



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## PUBLICATIONS

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### 3721 • High Performance Liquid Chromatography of Fatty Methyl Esters: Preparative Separations

C. R. Scholfield

Anal. Chem. 47(8): 1417-1420. July 1975

To separate small samples of fatty methyl esters and methyl ester fractions from hydrogenated fats a high-performance liquid chromatographic system with a column filled with a commercial packing of C<sub>18</sub> hydrocarbon bonded to silica and either acetonitrile or acetonitrile-water eluate is used with a differential refractometer detector. Methyl linolenate, linoleate, oleate, palmitate, and stearate may be separated with 100% acetonitrile. With 85 wt. % acetonitrile, methyl oleate, elaidate, and palmitate are separated; *cis*-15 octadecenoate occurs in the same peak as elaidate. Conjugated esters may be detected with an ultraviolet absorption detector, and conjugated linoleate isomers are eluted in the order *cis,trans*; *cis,cis*; and *trans,trans* with the *cis,trans* only slightly slower than *cis*-9,*cis*-12 linoleate. Saturated esters are eluted more slowly than monounsaturated esters with two more methylene groups. Esters from hydrogenated fats are separated into fractions suitable for further analysis.

### 3722 • Physiology of Sporeforming Bacteria Associated with Insects: Metabolism of *Bacillus popilliae* Grown in Third-Instar

*Popillia japonica* Newman Larvae

Grant St. Julian, Lee A. Bulla, Jr., and Richard S. Hanson<sup>1</sup>

(<sup>1</sup>University of Wisconsin, Madison)

Appl. Microbiol. 30(1): 20-25. July 1975

The timing and relative participation of concurrent pathways of carbohydrate metabolism as well as the extent of terminal respiratory activity were determined by radiorespirometry with <sup>14</sup>C substrates and by enzyme assays for vegetative and sporulating cells of the bacterium *Bacillus popilliae* cultured in whole, intact *Popillia japonica* (Japanese beetle) larvae. During vegetative proliferation, the pentose phosphate pathway predominates in the bacterial cells with minor involvement of the

Embden-Meyerhof-Parnas pathway. As the cells proceed through sporulation, pentose phosphate and Embden-Meyerhof-Parnas activity remains constant. No tricarboxylic cycle activity is evident during growth and sporulation of *B. popilliae*. The results demonstrate (1) predominantly aerobic metabolism for carbohydrate assimilation within in vivo sporulating cells, (2) a major contrast to the metabolism of other aerobic sporeforming bacteria that exhibit derepression of tricarboxylic acid cycle enzymatic activity at the onset of sporulation, and (3) no causal necessity of the cycle to *B. popilliae* sporogeny.

3723 • Changes in Microbial Population During Fermentation of Feedlot Waste with Corn

G. R. Hrubant

Appl. Microbiol, 30(1): 113-119, July 1975

A new process for recycling feedlot waste involves the fermentation of liquid from this waste combined with corn. Changes in the flora of this silage-like fermentation were followed. The fermentation was dominated by lactobacilli and yeasts, which initially constitute 1% or less of the natural flora. The species of yeasts and lactics involved were characterized. The fermentation has two phases. A single heterolactic species multiplied rapidly for the first 24 hours until it represented 95% of the lactobacilli and more than 90% of the total microflora. It displaced the betabacterium predominant among lactics of the original waste; the acid produced killed coliforms and other organisms in feedlot waste; and the acetic acid produced probably caused the death of the dominant native yeast *Trichosporon cutaneum* (de Beurm., Gougerot et Vaucher) Ota. The peak lactobacillus count remained constant [about  $2 \times 10^9$  organisms per gram (wet weight)] throughout the rest of the fermentation. Homolactics dominated the later phase and yeasts increased to  $9.5 \times 10^7$  organisms per gram (wet weight). At 6 days, a stable mixture of three lactobacilli was present, one streptobacterium, one thermobacterium, and one betabacterium. Similarly, yeasts stabilized as a mixture of two *Candida* sp. and one *Pichia* sp. The dominant species of lactics were characterized. Information on the sequence of microorganisms provides a basis for enhanced protein synthesis in the fermentation.

3724 • A Quantitative Method for Determination of Aflatoxin B<sub>1</sub> in Roasted Corn

Gail M. Shannon and Odette L. Shotwell

J. Assoc. Off. Anal. Chem. 58(4): 743-745, July 1975

Roasting aflatoxin-contaminated corn will reduce toxin levels. A quantitative analysis for aflatoxin in roasted corn has been developed by modifying a cleanup technique for green coffee extracts approved as



official first action by the Association of Official Analytical Chemists. A chloroform extract is partially purified on an activated magnesium silicate column, and thin layer chromatographic (TLC) plates are developed with methylene chloride-chloroform-isoamyl alcohol-formic acid (81+15+3+1). Recoveries average 101% and the sensitivity limit is 5 p.p.b. aflatoxin B<sub>1</sub>. A two-dimensional TLC procedure can also be used to separate the aflatoxins from background interferences.

3725 • Volatile Components from Tristearin Heated in Air

E. Selke, W. K. Rohwedder, and H. J. Dutton

J. Am. Oil Chem. Soc. 52(7): 232-235. July 1975

Tristearin was heated to 192° C. in air, and its volatile oxidation products were collected directly on a cooled (-60° C.) gas-liquid chromatography column. Subsequently, volatile products were separated by temperature programming up to 250° C. and identified by mass spectrometry. Methyl ketones and aldehydes were the major degradation products, along with minor amounts of monobasic acids, *n*-hydrocarbons, primary alcohols, and gamma lactones. Qualitative results indicated that all the fatty acid methylene carbon atoms are susceptible to oxidation. Since quantities of aldehydes and ketones were in excess of their taste threshold concentrations, thermally oxidized saturated fatty acids may be precursors of some odors and flavors associated with heated lipids.

3726 • Nucleophilic Addition of Hydrogen Sulfide to Methyl Oleate, Methyl Linoleate, and Soybean Oil

A. W. Schwab, L. E. Gast, and W. K. Rohwedder

J. Am. Oil Chem. Soc. 52(7): 236-239. July 1975

Hydrogen sulfide was added to methyl oleate, methyl linoleate, and soybean oil at -70° and 25° C. in the presence of boron trifluoride. Major reaction compounds were identified by gas-liquid chromatography and mass spectrometry. At -70° C. with a 200 molar ratio of hydrogen sulfide to ester, the reactions were complete in 4 hours. Primary reaction product from methyl oleate was methyl 9(10)-mercaptostearate. Methyl linoleate gave approximately equal amounts of methyl 9-(2-pentyl-1-thiolan-5-yl) nonanoate and methyl 8-(2-hexyl-1-thiolan-5-yl) octanoate. At 25° C. the reaction of methyl oleate and linoleate with hydrogen sulfide was less complete, and more side reactions were noted. When equimolar amounts of methyl oleate and methyl 9(10)-mercaptostearate were reacted in the presence of boron trifluoride at 25° C., a new compound was formed, bis[methyl *n*-octadecanoate 9(10)-yl] sulfide. The addition of liquid hydrogen sulfide to soybean oil at -70° C. in the presence of boron trifluoride yields a product which, upon saponification, acidification, and methylation analyzes by gas-liquid chromatography as approximately 52% thiolan, 27% mercaptostearate, 10% palmitate, 6% stearate, and 5% unidentified compounds.

3727 • Selective Hydrogenation of Soybean Oil: VII. Poisons and Inhibitors for Copper Catalysts

Sambasivarao Koritala

J. Am. Oil Chem. Soc. 52(7): 240-243, July 1975

Hydrogenation rates for the catalytic reduction of soybean oil with a copper-on-silica catalyst increased when the oil was re-refined and bleached in the laboratory. Purification of the re-refined and bleached oil by passage through alumina further enhanced hydrogenation rates. Since these observations suggested that poisons were present in the oil, the effect of minor components of soybean oil on the activity of copper catalysts was investigated.

Free fatty acids, monoglycerides, beta-carotene, phosphoric acid, sodium soaps, phosphatides, glycerine, choline, ethanolamine, water, pheophytin, and pyrrole all reduced hydrogenation rates when added to the oil. Organic sulfur added to the oil was a more effective catalyst inhibitor than inorganic sulfur added to the gas. Catalyst activity was affected adversely when iron was added to the oil as a soap or when deposited on the catalyst during its preparation. Squalene, copper soaps, and carbon monoxide had no influence on the activity of the catalyst. Aging of soybean oil also had no effect. There was no significant change in either selectivity or formation of *trans* or conjugated diene isomer when these additives were added to the oil.

3728 • Competitive Hydrogenation Rates of Isomeric Methyl Octadecadienoates

J. M. Snyder, C. R. Scholfield, T. L. Mounts,  
R. O. Butterfield, and H. J. Dutton

J. Am. Oil Chem. Soc. 52(7): 244-247. July 1975

Determination of the relative reaction rates of isomeric methyl octadecadienoates is possible by competitive reduction of a mixture containing an inactive diene and a radioactively labeled isomer. The hydrogenation rate of methyl *cis*-9,*cis*-12-octadecadienoate with platinum and nickel catalysts is compared to the hydrogenation rate of each of several isomers of methyl octadecadienoate, and the relative rate of the competitive hydrogenations is calculated by a digital computer. Methyl *cis*-9,*cis*-12 linoleate is reduced the most rapidly of all the dienes studied. The relative rates of the positional isomers tend to decrease with the increasing number of methylene groups between the double bonds, except when one of the double bonds is in the more reactive 15 position. Comparison of the geometric isomers shows that *trans,trans* diene is hydrogenated at a slower rate than *cis,cis* linoleate.

## 3729 • Kenaf Pulp--Soda vs. Sulfate

M. O. Bagby, R. L. Cunningham, and T. F. Clark  
Tappi 58(7): 121-123. July 1975

Chemical and physical characteristics of soda pulps from green, field-dried, and stored kenaf, as well as from kenaf bark and woody core were compared with those for sulfate pulps from the same five raw materials. In contrast to the long-recognized superiority of kraft or sulfate wood pulps over wood soda pulps, strength characteristics of kenaf soda pulps equalled those of kenaf sulfate pulps. Yields were comparable for either process. However, drainage of the kenaf soda pulps was slightly better than that of the kenaf sulfate pulps. These favorable results reinforce the concept of applying the sulfur-free soda technique to pulp kenaf and lower pollution.

## 3730 • Wet-Milling Properties of Corn After Field Shelling and Artificial Drying

C. Vojnovich, R. A. Anderson, and E. L. Griffin, Jr.  
Cereal Foods World 20(7): 333-335. July 1975

Extreme artificial drying of corn is detrimental to its wet milling by reducing yields of oil and starch, raising protein content of the starch, and changing past viscosity characteristics of the starch. Picker-sheller damage during harvest can also affect the recovery of prime milling products. There appears to be no correlation between test weight and the recovery of oil or starch.

## 3731 • Single-Kernel Analysis of Glutenin: Use in Wheat Genetics and Breeding

J. A. Bietz, K. W. Shepherd,<sup>1</sup> and J. S. Wall  
(<sup>1</sup>Waite Agricultural Research Institute, University of  
Adelaide, South Australia)  
Cereal Chem. 52(4): 513-532. July-August 1975

Because of the importance of glutenin proteins in determining wheat flour quality and dough properties, new methods were developed for examining glutenin's subunit composition in hexaploid and tetraploid varieties with known genetic differences. Glutenin was quantitatively extracted from single kernels, and its subunit composition observed by sodium dodecyl sulfate-polyacrylamide gel electrophoresis. In the variety Chinese Spring, the five highest molecular weight (MW) subunits (1: 133,000 MW; 2: 104,000 MW; 3: 93,000 MW; 4: 86,000 MW; and 5: 68,000 MW) are coded by the long arms of chromosomes 1B (subunits 2 and 3), 1D (subunits 1 and 4), and 4D (subunit 5). Analysis of genetic stocks which contain these markers for chromosomes related to quality may permit selection or development of improved varieties; half-kernels may be examined, and lines maintained by



growing germ ends. Hexaploid wheats were also compared with durum and derived tetraploid wheats, which lack the entire D genome. Most tetraploid wheats lack glutenin subunits 1, 4, and 5, but only subunit 4 is consistently absent, and in many varieties considerable variability occurs. Hexaploid varieties, however, are more uniform in glutenin subunit composition.

- 3732 • Microbial Polysaccharides. Information on Polysaccharide YB-4163  
[P. R. Watson, P. A. Sandford, K. A. Burton, M. C. Cadmus,  
and A. Jeanes]  
North. Reg. Res. Lab., U.S. Agric. Res. Serv., CA-NRRL-43,  
6 pp. August 1975 [Processed]

This product, from the research efforts of a team of microbiologists and carbohydrate chemists, is one of a series of polysaccharides made by fermentation processes developed at the Northern Laboratory. Information presented is preliminary and is subject to further confirmation and extension as current research progresses and as industrial evaluation justifies.

Polysaccharide YB-4163 possesses a unique constitution, and its properties make the polysaccharide suitable for industrial applications, because: (1) it is water soluble, (2) it forms flexible films, (3) it stabilizes oil-water emulsions, and (4) it stabilizes foams.

- 3733 • *cis*-5-Polyenoic Acids in *Larix leptolepis* Seed Oil  
R. D. Plattner, G. F. Spencer, and R. Kleiman  
Lipids 10(7): 413-416. July 1975

*cis*-5,*cis*-9-Octadecadienoic acid (2.7%) and *cis*-5,*cis*-9,*cis*-12-octadecatrienoic acid (24.9%) are present in the seed oil of *Larix leptolepis*. The double bond positions were identified by ozonolysis and by gas chromatography-mass spectrometry of methoxy derivatives. Small quantities of branched chain acids of various chain lengths were indicated by gas chromatography. The presence of C<sub>17</sub> and C<sub>19</sub> branched methyl esters was confirmed by gas chromatography-mass spectrometry.

- 3734 • Crystallographic Study of Tritetracosanoin  
T. D. Simpson and J. W. Hagemann  
J. Am. Oil Chem. Soc. 52(8): 303-306. August 1975

Crystallographic examination and computerized structural analysis of the X-ray powder diagram of tritetracosanoin show the molecule to be isomorphous with saturated triglyceride structures previously established. Cell dimensions are:  $a = 13.1$ ,  $b = 5.30$ ,  $c = 58.9$  Å;  $\alpha = 94$ ,  $\beta = 98$ ,  $\gamma = 99^\circ$ . Space group is  $P\bar{1}$ . The molecule is turned relative to the unit cell  $a$ -axis

likely because of acyl chain packing and end group plane interactions. Results indicate that the glycerol unit largely determines the lateral packing of saturated even-membered triglycerides. The method of structure solving indicates that saturated triglycerides can be studied by computerized structural analysis.

3735 • Substitution Reactions of Linoleic Acid Hydroperoxide Isomerase

D. D. Christianson and H. W. Gardner  
Lipids 10(8): 448-453. August 1975

Linoleic acid hydroperoxide isomerase was extracted from corn germ and partially purified by differential centrifugation. This enzyme catalyzed the isomerization of linoleic acid hydroperoxide ( $\text{R-CHOOH-CH}=\text{CH-CH}_2\text{-CH}=\text{CH-R}_1$ ) to the expected  $\alpha$ -ketol ( $\text{R-CHOH-CO-CH}_2\text{-CH}=\text{CH-R}_1$ ) and  $\gamma$ -ketol ( $\text{R-CH}_2\text{-CO-CH}=\text{CH-CHOH-R}_1$ ). Isomerase also catalyzed the substitution of various reagents at the carbon bearing the hydroperoxide group. These fatty acid products had the following functional groupings:  $\text{R-CHX-CO-CH}_2\text{-CH}=\text{CH-R}_1$  where X is either oleoyloxy, ethylthio, or methoxy resulting from the presence of oleic acid, ethanethiol, or methanol, respectively. A crude wheat germ extract containing both lipxygenase and isomerase enzymes reacted with linoleic acid to yield  $\alpha$ -ketols,  $\gamma$ -ketols, and a substitution product, the linoleoyloxy ester of  $\alpha$ -ketol. Characterization of these products from wheat germ enzymes showed that the substitution reaction was not unique to corn germ. Because anions of the reagents tested are typical nucleophiles, the substitution reactions may proceed by a nucleophilic mechanism as mediated by the isomerase enzyme.

3736 • *Taxus baccata* Seed Oil: A New Source of *cis*-5,*cis*-9-Octadecadienoic Acid

R. V. Madrigal and C. R. Smith, Jr.  
Lipids 10(8): 502-504. August 1975

Methyl esters prepared from the seed oil of the conifer *Taxus baccata* L. were found by gas-liquid chromatography to contain 12% of a component which, when isolated by preparative thin-layer chromatography and characterized by mass spectrometry, ozonolysis, and nuclear magnetic resonance, was identified as *cis*-5,*cis*-9-octadecadienoic acid.

- 3737\* • Growth-Promoting Effects of Fermented Soybeans for Broilers  
 C. C. Chah,<sup>1</sup> C. W. Carlson,<sup>1</sup> G. Semeniuk,<sup>1</sup> I. S. Palmer,<sup>1</sup>  
 and C. W. Hesseltine  
 (<sup>1</sup>South Dakota State University, Brookings)  
 Poult. Sci. 54(2): 600-609. March 1975

Four factorial experiments are here reported on the use of control and fermented soybeans in glucose-soybean diets for broiler chicks with varying protein levels (15, 17, and 19% and 13, 16, and 19%). Eleven strains of *Aspergilli* were used as cultures in these experiments, selected on the basis of a prior history of beneficial effects. Data obtained from 4-week growth studies revealed that feeding soybeans fermented with 10 of the 11 species gave significant ( $P < 0.05$ ) improvements in weight gain and feed efficiency. The responses were more pronounced with the low dietary levels of protein. No detrimental effect was shown from any of the cultures tested. Chemical analyses indicate that chicks fed the fermented soybean diets made better use of dietary nitrogen and dry matter. Carcass composition data show that the diets made with fermented soybeans produced chicks that were significantly ( $P < 0.05$ ) higher in protein and ash and lower in total lipids. Amino acid analyses suggest that the growth-promoting activity was largely due to a greater supply of the essential amino acids. Some vitamin synthesis by the fungi is a possibility.

- 3738 • Fungal Metabolites and Viral Replication in *Penicillium stoloniferum*  
 R. W. Detroy and P. E. Still  
 Dev. Ind. Microbiol. 16: 145-151. 1975

Little is known about the relationship between mycoviruses and fungal secondary metabolism. An in vivo replication system from *Penicillium stoloniferum* strain NRRL 5267 has been developed to test various fungal products and other antimetabolites against *P. stoloniferum* fast-moving virus (PsV-f) replication. Initially, cycloheximide was investigated in this test system because of its known antifungal and antiviral activity. Fractions of 48-hour mycelia were incubated in the presence of various metabolites, and after an additional 48-hour incubation period, the PsV-f content was measured in  $A_{260}$  units by polyacrylamide gel electrophoresis. Cycloheximide at 1, 5, and 10  $\mu\text{g/ml}$  blocked PsV-f replication 40, 75, and 95%, respectively, compared to untreated controls. At these levels fungal growth (biomass, dry weight) was unaffected. Therefore, cycloheximide proved to be a rather selective inhibitor of PsV-f replication. Mycophenolic acid, an antiviral metabolite produced by virus (-) strains of *P. stoloniferum* and *P. brevis-compactum*, at 300  $\mu\text{g/ml}$  blocked PsV-f replication by 25% but also reduced growth by 36%. Of the fungal metabolites tested only patulin and gliotoxin inhibited PsV-f replication.



- 3739 • Glutaminase Production by Bacteria  
G. E. N. Nelson, R. E. Peterson, and A. Ciegler  
Dev. Ind. Microbiol. 16: 385-390. 1975

About 100 strains of bacteria were analyzed for L-glutaminase, an enzyme known to have antitumor activity, after growth in shaken flasks on a medium containing 0.5% each of tryptone and yeast extract, as well as 0.1% each of dextrose and dibasic potassium phosphate. *Pseudomonas testosteroni* NRRL B-2611, which produced 0.46 international units (IU) of glutaminase activity per ml. of culture, was selected for intensive study. With this organism, tryptone and yeast extract had nearly equal effects on the yield of glutaminase, but no other protein-derived materials equaled them as adjuvants for production of this enzyme. Corn steep liquor stimulated growth but depressed enzyme yield. Growth and enzyme curves ran closely parallel, reaching a plateau between 10 and 12 hours. Glutaminase production was substantially the same at 24° C., 28° C., and 32° C. (0.34, 0.38, and 0.38 IU/ml., respectively) but decreased at 36° C. (0.18 IU/ml.). The pH optimum of partially purified glutaminase preparations from disrupted cells ranged from 7.5 to 8.0. Preliminary studies with gel filtration columns indicated a molecular weight around 100,000.

- 3740 • Bright Greenish-Yellow Fluorescence and Aflatoxin in Agricultural Commodities  
R. J. Bothast and C. W. Hesseltine  
Appl. Microbiol. 30(2): 337-338. August 1975

The corn milling industry has widely accepted the presence of bright greenish-yellow fluorescence under a black light as a presumptive indicator of aflatoxin (a poison produced by the mold *Aspergillus flavus*). This test was applied to wheat, oats, barley, rice, coconut, white corn, yellow corn, peanuts, sorghum, and soybeans, and evaluated in the laboratory. Our study supported the use of bright greenish-yellow fluorescence as a presumptive test for aflatoxin in wheat, oats, barley, corn, and sorghum.

- 3741 • Halogenated Allyl Glucoside-Based Flame-Retardant Urethane Foam  
Carl A. Wilham, Felix H. Otey, Charles L. Mehltretter, and Charles R. Russell  
Ind. Eng. Chem. Prod. Res. Dev. 14(3): 189-192. September 1975

The free radical addition of halohydrocarbons to propoxylated allyl glucoside offers a feasible route for incorporating high levels of halogens into polyethers. Urethane foams made from these polyethers exhibit normal physical properties and a higher level of flame resistance than might be expected from the amount of halogen added.

- 3742 • Scanning and Transmission Microscopy of Dough and Bread  
 U. Khoo, D. D. Christianson, and G. E. Inglett  
 Baker's Dig. 49(4): 24-26. August 1975

Flour, newly mixed and fermented doughs, and bread crumb from a good bread wheat variety, Bison, were studied by optical and electron microscopy. Scanning electron microscopy showed external characteristics of protein matrix and starch relationships to be dramatically different in flour, doughs, and bread crumb. Transmission electron microscopy revealed details of structure in the protein matrix. Numerous osmophilic particles or inclusions, which contain lipoproteins, were observed in all stages of dough development and baking. The size, as well as alignment, of osmophilic inclusions was changed when flour was mixed into dough. Baking does not appear to change protein ultrastructure drastically. Some starch membranes survive dough development and baking.

- 3743 • Blister Resistance of Linseed Oil Paints from  
 Treated Pigments  
 R. L. Eissler  
 J. Paint Technol. 47(607): 50-55. August 1975

The influence of certain surface treatments for zinc oxide or titanium dioxide pigments and the effect of heat-bodying linseed oils on blister resistance of paints is examined. Paints were formed from treated or untreated pigments in bodied and in unbodied linseed oils. Blister resistance of paints after application to western red cedar panels was measured on a blister box. Experimental paints, each with the same control, were tested in a two-coat test system. Data were taken from life-sized photographs according to ASTM Method D714-56 and also by counting and determining blister size. Quantitative data from the latter method are subjected to a four-way analysis of variance for significant differences. Ratios of blistering of experimental paint to that for control paint on the same panel are used in an attempt to minimize effects caused by individual wood panels. Pigment type, pigment surface treatment, and oil body all appear to influence blister resistance of test paints.

- 3744 • Fermentation of Whole Feedlot Waste and Isolated Feedlot  
 Waste Fiber with *Trichoderma viride* in Submerged Culture  
 Harold L. Griffin, Tsuneo Kaneshiro, Benjamin F. Kelson,  
 and James H. Sloneker  
 In "Symposium on Enzymatic Hydrolysis of Cellulose," held at  
 Aulanko, Finland, March 12-14, 1975, pp. 419-431. 1975

When crude feedlot waste (FLW) fiber is separated from the whole FLW, *Trichoderma viride* ferments it better. After separation, solids concentration can be increased from 2.5% for whole waste to 16.7% for the isolated fibers with a concomitant increase in the rate of fiber decomposition. Approximately 65% of the cellulose and hemicellulose is utilized.



When FLW fiber isolated from fresh FLW is fermented, the total reduction of solids is 38%. Of the remaining solids, mycelial mass increases the crude protein of the fermented residue at least 30-40%. Both solids reduction and crude protein increase improve the feed additive potential of FLW fiber. Yields of cellulase enzyme are lower than those in fermentations of whole FLW with *T. viride* but are comparable to those reported for this organism grown on refined cellulose.

3745 • Preparation of Protein Concentrate from Normal and High-Protein Wheats

Y. Victor Wu and Kenneth R. Sexson

J. Agric. Food Chem. 23(5): 903-905. September-October 1975

An alkaline extraction process was developed to produce protein concentrates from high- and normal-protein wheats. Different solvents, various pH values, wheat-to-solvent ratios, and particle size of wheat were studied. Optimum extraction was at pH 10.8 in 0.03 *N* sodium hydroxide with 100 g. of wheat per 600 ml. of solvent. After centrifugation each of two alkaline extractions was adjusted to pH 6 to yield a precipitate and a supernatant. Bran was removed from starch and protein by screening the second alkaline dispersion. The protein content (nitrogen X 5.7) of the concentrate varied between 83 and 92%, depending on particle size and protein content of the wheat used, and accounted for 52 to 64% of the total wheat protein. Prime starch (0.3% protein) was also produced in good yield. Higher yields of protein were obtained from wheat containing higher protein.

3746 • Composition and Properties of Protein Concentrate from Normal and High-Protein Wheats

Y. Victor Wu and Kenneth R. Sexson

J. Agric. Food Chem. 23(5): 906-909. September-October 1975

Protein concentrates and byproducts produced from ground wheat were analyzed for amino acid composition, protein, starch, fat, fiber, ash, and various neutral carbohydrates. The concentrates contained from 83 to 92% protein (nitrogen X 5.7), 2.1 to 2.6 g. of lysine, 2.5 to 3.5 g. of total sulfur amino acids per 16 g. of nitrogen, 1.5 to 7.4% fat, 0.9% ash, and from 1.8 to 3.9% total carbohydrate. Protein concentrate had a nitrogen solubility of 97% at pH 2.5 and a minimum solubility of 5.5% at pH 6.2, a hydration capacity of 2.6, had an emulsion stability of 63%, and formed a reasonably strong and elastic gluten ball.

## 3747\* • Microscopy in Coatings and Plastics Research

L. H. Princen

*In "Applied Polymer Science," eds. J. K. Craver and R. W. Tess, chap. 9, pp. 92-99. 1975*

Fifty years ago only optical microscopy was available for coatings and plastics research. Although several modes of operation were already developed, the inherent limitations in resolution and depth-of-focus made the optical microscope less suitable for polymer research than for medical and biological purposes. After World War II, the transmission electron microscope (TEM) and special specimen preparation techniques were sufficiently developed to become useful in polymer research. Limited depth-of-field and complicated specimen preparation continued to be major drawbacks to TEM research of coatings and plastics. In 1965, the scanning electron microscope (SEM) was commercially introduced and almost overnight was accepted universally; the simplicity of specimen preparation and the spectacular image quality particularly made this tool popular. Its impact on coatings and plastics has been great already. Its major drawback has been cost of equipment and operation. The key publications that cover the history of microscopy and its impact on polymer research are cited in this review. Also peripheral techniques, such as electron microprobe, X-ray, and electron spectroscopy, are treated briefly, as well as some desired developments that can be expected in the future.

## 3748\* • Theories of Solvency and Solution

E. B. Bagley

*In "Applied Polymer Science," eds. J. K. Craver and R. W. Tess, chap. 43, pp. 632-643. 1975*

Complex polymer-solvent systems commonly occur in the coatings and plastics industry. These complex systems have led to the successful industrial development of empirical and semi-quantitative treatments of the thermodynamics of phase separation. These empirical correlations and predictive procedures rest on a firm physical-chemical basis. By considering factors usually neglected in treating phase-separation problems, particularly the volume change on mixing, these empirical procedures can be put on a completely quantitative basis. The inter-comparison of practical and theoretical developments of recent years permits some estimate of the reliability of the empirical and semi-quantitative procedures to be made wherever fundamental data needed for a quantitative treatment are not available.



- 3749 • Differential Inhibition by  $\beta$ -Exotoxin of Vegetative- and Sporulation-Specific Ribonucleic Acid Polymerases from *Bacillus thuringiensis* Cells  
Donovan E. Johnson, Lee A. Bulla, Jr., and Kenneth W. Nickerson  
Spores 6: 248-254. 1975

Deoxyribonucleic acid (DNA)-dependent ribonucleic acid (RNA) polymerase isolated from cells of *Bacillus thuringiensis* var. *thuringiensis* is inhibited by  $\beta$ -exotoxin, a phosphorylated adenine nucleotide derivative produced extracellularly by a variety of *B. thuringiensis* strains. A concentration of  $1.25 \times 10^{-4}$  M exotoxin caused 50% inhibition of the vegetative polymerase activity isolated during exponential growth. The same exotoxin concentration resulted in only a 30% inhibition of the sporulation-specific polymerase isolated approximately at  $t_4$ . The differential inhibition of transcription rates of the two enzymes in the presence of exotoxin was apparent with *B. subtilis* or calf thymus DNA as template, but no variation in transcription rates was evident with a synthetic template [poly(deoxyadenylate-deoxythymidylate)]. No significant difference in sedimentation characteristics between the vegetative- and sporulation-specific enzymes could be detected in glycerol density gradients. Delayed addition of exotoxin to a preparation actively synthesizing RNA caused an immediate and complete cessation of RNA synthesis by either enzyme. The data indicate that exotoxin is a positive modifier of *B. thuringiensis* RNA polymerase in vitro and may function during sporulation in this organism.

- 3750 • Publications and Patents of the Northern Regional Research Laboratory, January-June 1975  
North. Reg. Res. Lab., ARS, USDA, 58 pp. [August 1975]

- 3751 • Graft Copolymers of Dextrin and Polyacrylamide as Filler Retention Aids  
H. D. Heath, G. F. Fanta, A. J. Ernst, B. T. Hofreiter, and R. C. Burr  
Svensk Papperstidning 78(13): 488-491. September 1975

Acrylamide was grafted to dextrin by simultaneous  $\gamma$ -irradiation in water. Dextrin graft copolymers containing about 50% polyacrylamide by weight were evaluated as retention aids on a pilot fourdrinier in the production of filled bond paper. Effectiveness increased as graft molecular weight increased. Introduction of carboxylate groups did not affect performance. Dextrin graft copolymers were more effective than physical mixtures of polymer components (dextrin and polyacrylamide). Wet-end additions of blends of dextrin graft polymer with unmodified starch improved both paper strength and filler retention.

3752\* • Induction of Fescue Foot Syndrome in Cattle by Fractionated Extracts of Toxic Fescue Hay

M. Williams,<sup>1</sup> S. R. Shaffer,<sup>1</sup> G. B. Garner,<sup>1</sup> S. G. Yates,  
H. L. Tookey, L. D. Kintner,<sup>1</sup> S. L. Nelson,<sup>1</sup> and  
J. T. McGinity<sup>1</sup>

(<sup>1</sup>University of Missouri, Columbia)

Am. J. Vet. Res. 36(9): 1353-1357. September 1975

Tall fescue (*Festuca arundinacea* Shreb) hay from a source known to cause "fescue foot" in grazing cattle was extracted with 80% ethanol. The ethanolic extract was further refined and fractionated into cation, anion, and neutral fractions by ion-exchange chromatography. The cation fraction was partitioned with alkaline-chloroform to give chloroform-extractable cation and residual cation fractions. All fractions plus the crude ethanolic extract were assayed for toxic activity by intraperitoneal injection into 12 calves (weighing 152.4 to 241.3 kg. each) over a 14-day period. Clinical signs of fescue foot were observed on the fifth day in calves given the anion and crude ethanolic extracts. Lameness, swelling, and reddening of the rear coronary bands, discoloration of the tip of the tail, and other signs of fescue foot were seen. Microscopically, coronary bands and tail tips of affected calves had blood vessels with thick walls and small lumens.

3753 • Free Fatty Acids Identified as Antitryptic Factor in Soybeans Fermented by *Rhizopus oligosporus*

Hwa L. Wang, E. W. Swain, L. L. Wallen, and C. W. Hesseltine  
J. Nutr. 105(10): 1351-1355. October 1975

The trypsin inhibitory activity observed in cooked soybeans fermented by *Rhizopus oligosporus* (fungus used in tempeh fermentation) has been examined. The active compounds have now been isolated by ethanol extraction and thin-layer chromatography and have been identified as free fatty acids by infrared spectroscopy and gas-liquid chromatography. Oleic, linoleic, and linolenic acids are primarily responsible for the increased trypsin-inhibiting activity of cooked soybeans after fermentation.

These free fatty acids are liberated from oil in the soybeans by fungal lipase, and they differ from other reported soybean trypsin inhibitors that are protein in nature. Free fatty acids have previously been reported to inhibit various enzymes, such as glycolytic, glyconeogenic, lipogenic, and also proteolytic. Their effect appears to be a non-specific type of inhibition. Further studies are required to determine their physiological relevance, if any.

## 3754\* • Protein Sweeteners

G. E. Inglett

In "The Chemistry and Biochemistry of Plant Proteins,"  
eds. J. B. Harborne and C. F. Van Sumere, Proc. Phytochem.  
Soc. Symp., Ghent, Belgium, September 4-6, 1973, chap. 9,  
pp. 265-280. 1975

Sweetness is a gustatory response evoked by substances on a sweet taste bud which transmits a message to the brain indicating sweet taste. An unusual source of sweet taste is present in a West African berry known as miracle fruit (*Synsepalum dulcificum*). This fruit possesses a taste-modifying substance that causes sour foods--e.g., lemons, limes, or grapefruit--to taste sweet. Inglett and his associates found the active principle to be macromolecular and assumed it to be glycoprotein, an assumption later confirmed by others. Until this time, only small molecules were considered sweet-invoking substances. Now macromolecules are considered capable of participating in taste perception.

The intense sweetness of the fruit of *Dioscoreophyllum cumminsii*, called the serendipity berry, was also revealed to be a macromolecule. In 1972, this sweetener was found to be a protein. Also in 1972, the intensely sweet principle of *Thaumatococcus daniellii* fruit was reported to contain two proteins having intense sweetness.

Since intensely sweet protein sweeteners act directly on taste buds as a probe, a peptide linkage analogous to the aspartic acid sweeteners may be partly responsible for their sweetness. As an extension of the probe theory, chemical structures of the intense glycosidic sweeteners--stevioside, glycyrrhizin, osladin, and the dihydrochalcones--were examined. The obvious similarity among these intense sweeteners is the occurrence of a (1'→2)-oxygen-linked disaccharide attached to an aglycone.

3755\* • Biosynthesis of Fatty Acids During Germination and Outgrowth of *Bacillus thuringiensis* Spores

Lee A. Bulla, Jr.,<sup>1</sup> Kenneth W. Nickerson, Timothy L. Mounts,  
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Spores 6: 520-525. 1975

Selective synthesis of branched- and normal-chain fatty acids occurred during germination and outgrowth of *Bacillus thuringiensis* spores. Iso-C<sub>13</sub>, iso-C<sub>14</sub>, and iso-C<sub>16</sub> were the first fatty acids to appear upon germination, their formation occurring prior to protein synthesis. Afterwards, normal-C<sub>14</sub>, normal-C<sub>16</sub>, anteiso-C<sub>13</sub>, and anteiso-C<sub>15</sub> acids were synthesized maximally. During septum formation, anteiso-C<sub>17</sub> acid appeared, and there was a resurgence in iso-C<sub>16</sub> acid synthesis. Throughout outgrowth, iso-C<sub>15</sub> and iso-C<sub>17</sub> were metabolized uniformly, whereas the other acids exhibited peak synthesis.



- 3756 • Soybean Soapstock Utilization: Fatty Acid Adducts with Ethylene and 1-Butene  
R. E. Beal, L. L. Lauderback, and J. R. Ford  
J. Am. Oil Chem. Soc. 52(10): 400-403. October 1975

The relation of certain reaction variables to yield was investigated in the preparation of ethylene and 1-butene Diels-Alder adducts with alkali-conjugated linoleic and linolenic acid soaps derived from soybean oil soapstock. Adduct yields generally increased with pressure at the 295° C. reaction temperature. Maximum yields obtained with fatty acids derived from soapstock were about 80% of theory with ethylene and 40% of theory with 1-butene. Purification of adduct methyl esters by vacuum fractional distillation gave adducts with more than 95% purity. Ethylene adduct amides show promise as antiblock agents for plastic film.

- 3757 • Degradation of Lipids and Glucosinolates in Dehulled Crambe Seed During Storage  
E. C. Baker, G. C. Mustakas, and J. E. McGhee  
J. Am. Oil Chem. Soc. 52(10): 404-406. October 1975

Crambe as harvested contains seed accompanied by about one-third hull, a fraction of little commercial value that can be removed during processing. If the hull were removed before shipping, considerable transportation costs could be saved. Because little was known about the quality of crambe seed after dehulling and storage, decomposition of the glucosinolates in dehulled seed after storage was studied, as well as changes in color, free fatty acids, peroxide value, and ease of hydrogenation of the oil fraction. While not so stable as seed plus pericarp (hull), dehulled crambe seed can be stored for at least 3 months at temperatures up to 110° F. at ambient moisture (6.3%). However, at 14% moisture, decomposition was significant after storage for 2 weeks at both ambient (77° F.) and elevated (110° F.) temperatures. Inactivation of the endogenous enzyme after dehulling extends storage life to 3 months at either moisture level at ambient temperatures.

- 3758 • Effects of Feeding Formaldehyde Treated, Full Fat Soybean Flours on Milk Fat Polyunsaturated Fatty Acids  
J. Bitman,<sup>1</sup> T. R. Wrenn,<sup>1</sup> D. L. Wood,<sup>1</sup> G. C. Mustakas, E. C. Baker, and W. J. Wolf  
(<sup>1</sup>Biochemistry Laboratory, ARS, USDA, Beltsville, Maryland)  
J. Am. Oil Chem. Soc. 52(10): 415-418. October 1975

Raw, commercial, and extrusion cooked full-fat soy flours were treated with formaldehyde and then fed to dairy cows. This treatment protected the polyunsaturated fats of the soy from hydrogenation by microbial action in

the cow's rumen. With all of these materials, higher than usual amounts of polyunsaturated fats were incorporated into the milk. In a screening test limited to one cow, an advantage as measured by milk yield, fat yield, protein, solids-not-fat, and increased milk fat C18:2 was seen for the formaldehyde-treated, full-fat soy flour. The percentage of linoleic acid more than doubled in the milk fat of cows receiving the protected products. Only very slight quantities of formaldehyde (0.1-0.2 p.p.m.) were found in the milk. The efficiency of transfer of the C18:2 from the feed to milk was about 37%. This represented a marked improvement over previous trials in which we fed expensive safflower oil-casein-formaldehyde supplements.

3759 • Paper-Chromatographic Separation of [ $^3\text{H}$ ]Borohydride-Reduced Hexoses for Quantitative Determination of Polysaccharide Composition

Clarence A. Knutson

Carbohydr. Res. 43(2): 225-231. September 1975

An improved procedure for determination of composition of heteropolysaccharides by radiochromatography of sodium [ $^3\text{H}$ ]borohydride-reduced hydrolyzates has been developed. The method is accurate and sensitive, is less cumbersome and more reliable than similar procedures currently in use, and permits, for the first time, compositional analysis of hydrolyzates containing both hexoses and uronic acids in a single radiochromatographic procedure.

3760\* • Altered Feathering of Chicks Caused by T-2 Toxin

R. D. Wyatt,<sup>1</sup> P. B. Hamilton,<sup>1</sup> and H. R. Burmeister  
(<sup>1</sup>North Carolina State University, Raleigh)

Poult. Sci. 54: 1042-1045. October 1975

Dietary T-2 toxin (0, 1, 2, 4, 8, and 16  $\mu\text{g./g.}$ ) was fed to four groups of 10 chickens at each treatment level from hatching until 3 weeks of age. Growth inhibitory levels (4, 8, and 16  $\mu\text{g./g.}$ ) caused abnormal feathering which appeared dose related. The chickens were sparsely covered with short feathers protruding at odd angles in comparison to controls. There were few feathers at the base of the neck, on the anterior dorsal surface of the wing, and on the side and back adjacent to the tail. Feather tips frequently were constricted and bent downward while the quill could have a reverse curve. A literature survey suggests that T-2 toxin may cause this effect through a nutritional imbalance.

- 3761 • Positional Specificity of  $\gamma$ -Ketol Formation from Linoleic Acid Hydroperoxides by a Corn Germ Enzyme  
 H. W. Gardner, R. Kleiman, D. D. Christianson, and D. Weisleder  
*Lipids* 10(10): 602-608. October 1975

We have shown unequivocally that the positional specificity of  $\gamma$ -ketol formation by a corn germ enzyme was different from that observed previously by others with an alfalfa seedling enzyme. When the pure positional isomers of linoleic acid hydroperoxide served as substrates, the corn germ enzyme formed one of two  $\gamma$ -ketols: 12-oxo-9-hydroxy-*trans*-10-octadecenoic acid from 13-hydroperoxy-*cis*-9,*trans*-11-octadecadienoic acid (99+% pure) and 10-oxo-13-hydroxy-*trans*-11-octadecenoic acid from 9-hydroperoxy-*trans*-10,*cis*-12-octadecadienoic acid (96% pure). Also isolated from these reactions was one of two  $\alpha$ -ketols commonly found as a result of catalysis by linoleic acid hydroperoxide isomerase: 12-oxo-13-hydroxy-*cis*-9-octadecenoic acid from the 13-hydroperoxide and 10-oxo-9-hydroxy-*cis*-12-octadecenoic acid from the 9-hydroperoxide. Evidence is offered that  $\gamma$ -ketol formation is catalyzed by linoleic acid hydroperoxide isomerase, the same enzyme responsible for  $\alpha$ -ketol production.

- 3762 • Positive Displacement Holder for Critical Point Drying of Small Particle Materials  
 F. L. Baker and L. H. Princen  
*J. Microsc.* (Oxford) 103(Pt. 3): 393-401. April 1975

A specimen holder has been designed specifically to contain small particulate matter, such as microorganisms, fibers, or powders, during the fluid exchange cycle and final evaporation step of the critical point drying process. The mode of fluid exchange is positive displacement rather than diffusion. The holder can accommodate up to ten different samples without loss of material or cross-contamination during the dehydration process. The samples are separated by silver membrane filters, and fluid exchange is accomplished in minimal time by only slight positive pressure. Experiments with soluble dyes have shown that any fluid can be displaced completely with another in less than 2 minutes, whereas diffusion requires more than 30 minutes. The holder and individual capsules have been tested with different materials and a standard water/ethyl alcohol/amyl acetate/carbon dioxide fluid sequence. This paper describes the design, production, and testing of the holder and capsules, and presents selected examples of results.



3763\* • Protein Quality in Cereals Evaluated by Rapid Estimation of Specific Protein Fractions

Jerrold W. Paulis and Joseph S. Wall

In "Protein Nutritional Quality of Foods and Feeds. Part 1. Assay Methods--Biological, Biochemical, and Chemical," ed. Mendel Friedman, chap. 19, pp. 381-402. 1975

The content of lysine (the first limiting amino acid nutritionally in cereals) varies considerably among the different solubility classes of cereal proteins. Thus the proportion of lysine-deficient protein to total protein may serve as an index of protein nutritional value in cereals. In normal and high-lysine corns an inverse correlation ( $-0.87$ ) was established between total lysine content of the grain and the amount of zein (a protein low in lysine). Zein was precipitated from 70% ethanol-0.5% sodium acetate extracts of the corn meal by addition of salt solution and was measured turbidimetrically. This simple, rapid, and inexpensive procedure was used successfully to estimate lysine in large numbers of corn samples varying in genetic background, composition, and physical properties. It is a useful screening tool in breeding programs for vitreous *opaque-2* high-lysine corn. Variation in lysine content of the glutelin protein is a factor reducing accuracy of the method. An alcohol-soluble fraction of reduced glutelin has low lysine content. Extracting meals with 70% ethanol-0.5% sodium acetate containing 0.1 M mercaptoethanol yields this protein as well as zein. Turbidimetric analyses of total protein in this extract give an improved negative correlation with lysine content of the grain. Since nutritional value of protein in other grains such as sorghum and barley is also reduced by their prolamine and alcohol-soluble glutelin content, the method can be extended to them.

3764\* • Chemical and Biological Evaluation of Maize Protein Quality: Current Issues and Problems

Joseph S. Wall and Jerrold W. Paulis

In "High-Quality Protein Maize," Proc. CIMMYT-Purdue Symp. Protein Quality in Maize, El Batan, Mexico, December 4-7, 1972, Part VII, pp. 281-290. 1975

The need for improved methods of evaluating the nutritional quality of maize protein by chemical and biological procedures has developed as a result of the expansion of efforts to breed higher yielding high-lysine maize hybrids having superior kernel characteristics. Success in these efforts will permit high-lysine grain to enter market channels and to be processed for foods. Simple analytical methods will be required to distinguish high-lysine maize and ensure that its protein quality is maintained during processing. Fundamental information on the nature of corn proteins is being obtained as an aid in attaining these goals. For example, a major maize protein, glutelin, which constitutes the matrix

protein within the endosperm cells, consists of different types of subunits held together by disulfide bonds. The lysine composition of the protein is influenced by its subunit composition. While many methods of determining protein quality of maize depend on varied types of lysine analysis, rapid procedures are being developed based on properties of the grain proteins. A simple determination of zein in maize extracts by turbidimetric analysis gives a good negative correlation with lysine. Minimal heating of products of high-lysine maize or maize germ is required to prevent loss of biological value. In contrast to the expeller-processed product, germ that is extracted with solvent at low temperature retains high nutritive value.

- 3765 • Relationships of *Actinomycetales* to *Chytridiales*, *Hyphochytridiales*, *Blastocladales*, and *Oomycetales*  
 Thomas G. Pridham and A. J. Lyons  
 In "Proceedings of the First Intersectional Congress of IAMS. Vol. 1. Comparative Microbiology," ed. Takezi Hasegawa, pp. 3-18. 1975

Our literature survey and laboratory experiments clearly point out the nonrelationship of *Actinomycetales* and lower Phycomycetes on morphological and genetic bases. A possible area of similarity lies in physiological parameters, such as the presence of the DAP pathway to lysine, or other physiological activities common to many kinds of organisms. Beyond that, the only other similarities appear to be their hyphal nature and the presence of motile forms. Conceivably the *Actinomycetales* might have evolved independently, but in a fashion similar to the Phycomycetes. Both groups may well have evolved from aquatic motile ancestors to terrestrial nonmotile forms.

- 3766\* • Introduction [Part III. Mycotoxins]  
 Alex Ciegler  
 In "Microbiology--1975," ed. David Schlessinger, Part III, Mycotoxins, p. 343. 1975
- 3767\* • Mycotoxin Synergism  
 E. B. Lillehoj and A. Ciegler  
 In "Microbiology--1975," ed. David Schlessinger, Part III, Mycotoxins, pp. 344-358. 1975

This review summarizes the research observations of toxic synergism in test animals: specifically, the effects mediated by the interactions of mycotoxins with other chemical compounds and physical processes. Results

of particular interest to mycotoxicologists are examined, including mycotoxin-mycotoxin synergism, mycotoxin-xenobiotic interactions, nutritional modification of toxic effects, and the relationship of the hormonal status of test animals to the toxic manifestations of specific fungal metabolites. One section presents extensively the possible mechanisms of mycotoxin synergism. Implications are considered of the drug-metabolizing function of liver microsomes in processing toxic fungal metabolites and the association of the activity to toxic synergism. Several conceptual models are given to explain the relationship between the function of liver microsomes and expression of toxic symptoms.

- 3768 • Aflatoxin Occurrence in 1973 Corn at Harvest. I. A Limited Survey in the Southeastern U.S.  
 E. B. Lillehoj, W. F. Kwolek,<sup>1</sup> G. M. Shannon, O. L. Shotwell, and C. W. Hesseltine  
 (<sup>1</sup>Biometrician, North Central Region, ARS, USDA, Peoria, Illinois)  
 Cereal Chem. 52(5): 603-611. September-October 1975

Freshly harvested corn (18% moisture average) from the 1973 crop was examined for *Aspergillus flavus*-induced bright greenish-yellow (BGY) fluorescence and for aflatoxin. The survey was carried out in a limited area (2,000 square miles) of the southeastern United States. In a previous investigation, the Food and Drug Administration had identified aflatoxin in a shipment of corn grown in the region in 1972. In the current study, 297 samples of shelled corn (10 lb. each) were collected; 184 samples were collected at field sites and 113 at elevator delivery points during harvest. Corn samples were dried to 13% moisture or below within 6 hours (mean time) after collection. Of the samples, 152 exhibited BGY fluorescence in whole and ground fractions and 94 contained aflatoxin B<sub>1</sub> above 20 p.p.b. Evidently aflatoxin contamination of corn occurred before harvest. Preliminary observations indicated a relationship between insect damage and presence of the toxin. However, a clear cause-effect association between insect activity and *A. flavus* infection was not established.

- 3769 • Aflatoxin Occurrence in Some White Corn Under Loan, 1971. III. Association with Bright Greenish-Yellow Fluorescence in Corn  
 O. L. Shotwell, M. L. Goulden, A. M. Jepson, W. F. Kwolek,<sup>1</sup> and C. W. Hesseltine  
 (<sup>1</sup>Biometrician, North Central Region, ARS, USDA, Peoria, Illinois)  
 Cereal Chem. 52(5): 670-677. September-October 1975

Studies were made on the relation between the occurrence in white corn samples from 1,283 truckloads of Commodity Credit Corporation corn delivered



at an elevator in southeastern Missouri of bright greenish-yellow (BGY) fluorescence under ultraviolet light (365 nm) and the presence and levels of aflatoxin. Although numbers of BGY fluorescing particles were related to levels of aflatoxin, the BGY fluorescent test could not be used to determine levels of toxin. Of the 10-pound whole-kernel corn samples having at least one BGY fluorescing corn particle or kernel, 55% contained measurable aflatoxin: 28% contained more than 20 p.p.b. toxin, the present Food and Drug Administration guideline. The analytical method was sensitive to 1-3 p.p.b. aflatoxin. Of the corn samples that had more than 20 fluorescing kernels and particles, 95% were contaminated with aflatoxin. Sixty-five percent of them had more than 20 p.p.b. toxin. Cracking corn before ultraviolet light (365 nm) inspection revealed more samples with BGY fluorescence, and 19% of these contained more than 1-3 p.p.b. aflatoxin.

- 3770 • Mycotoxins in Hot Spots in Grains. I. Aflatoxin and Zearalenone Occurrence in Stored Corn  
O. L. Shotwell, M. L. Goulden, R. J. Bothast, and  
C. W. Hesseltine  
Cereal Chem. 52(5): 687-697. September-October 1975

A hot spot that developed *Aspergillus flavus* growth in a bin of corn in central Illinois during warm weather was investigated for mycotoxins. High levels of aflatoxin (1,000-1,700 p.p.b.) were detected in samples collected near the center of the hot spot defined by visible *A. flavus* sporulation. The location of the hot spot indicated that the moisture necessary for mold growth and aflatoxin formation could have come from rains blown through an open window. Corn collected at all depths from seven locations and probe samples taken under the hot spot were assayed for aflatoxin and zearalenone. Aflatoxin was not detected in samples collected farthest from the window. Zearalenone was detected in some of the samples collected, but it was not confined to any one part of the bin. The corn had been in the field an unusually long time before harvest because of cold, wet weather. Individual kernels selected from locations likely to contain aflatoxin and zearalenone were assayed for these mycotoxins. Even in kernels connected with *A. flavus* mycelia, aflatoxin-free kernels occurred adjacent to highly contaminated kernels. Of 140 kernels analyzed for both aflatoxin and zearalenone, 16 contained aflatoxin (260-38,000 p.p.b.  $B_1 + B_2$ ) and 12 had zearalenone (9,000-1,700,000 p.p.b.). In no kernel were both aflatoxin and zearalenone detected.

- 3771\* • Crystal and Molecular Structure of the One to One Complex of Rotenone and Carbon Tetrachloride  
 Satish K. Arora,<sup>1</sup> Robert B. Bates,<sup>1</sup> Raymond A. Grady,<sup>1</sup> and Norman E. Delfel  
 (<sup>1</sup>University of Arizona, Tucson)  
 J. Am. Chem. Soc. 97(20): 5752-5755. October 1975

The crystal structure of the 1:1 complex of the natural insecticide 7-rotenone and carbon tetrachloride ( $C_{23}H_{22}O_6 \cdot CCl_4$ ) becomes the first rotenoid structure to be solved by X-ray techniques. The space group is orthorhombic,  $P2_12_12_1$ . Cell dimensions are  $a = 21.137$  (7),  $b = 12.861$  (5),  $c = 9.516$  (3) Å and  $Z = 4$ . The structure has been refined to  $R = 0.063$ . Three of the four chlorine atoms in the carbon tetrachloride molecule are disordered. The results confirm the 6aS, 12aS, 5'R configurations deduced for rotenone by Büchi and coworkers and the V-shaped conformation found for rotenone in solution by Carlson and coworkers. The packing consists of stacks of nested V's of rotenone molecules, with  $CCl_4$  molecules filling the tunnels between the stacks. The failure of some rotenoids with six-membered E rings to crystallize is apparently due to steric inhibition of this type of stacking.

- 3772 • Effects of Refining Operations on Cereals  
 George E. Inglett  
 In "Nutritional Evaluation of Food Processing," 2nd ed., eds. Robert S. Harris and Endel Karmas, chap. 8, pt. 1, pp. 13-158. Westport, Conn. 1975

All cereal grains contain starch as the principal component. The second highest component of cereal grains is protein. Both content and nutritive value of cereal protein vary widely and depend particularly on seed heredity and environment during cultivation and harvest. Improvement in cereal protein quality and quantity has been a major thrust in plant breeding since the discovery in 1964 by E. T. Mertz and his coworkers at Purdue University that *opaque-2* corn (high lysine) had a protein composition which provides considerably better nutrition than ordinary corn. Besides carbohydrate and protein, this chapter covers the other nutrients found in wheat, corn, rice, oats, barley, and rye.

- 3773\* • Effects of Refining Operations on Legumes  
 Walter J. Wolf  
 In "Nutritional Evaluation of Food Processing," 2nd ed., eds. Robert S. Harris and Endel Karmas, chap. 8, pt. 2, pp. 158-187. Westport, Conn. 1975

Processing methods and refining operations markedly influence the chemical composition and nutritional properties of soybeans, peas, and beans.

### 3774\* • Natural Toxicants in Legumes and Oilseeds: Identification and Removal

Joseph J. Rackis

In "Food Safety," ed. G. R. Ammerman (chairman, IFT Short Course Committee), 1975 IFT Short Course sponsored by Inst. Food Technol., pp. 19-48. 1975

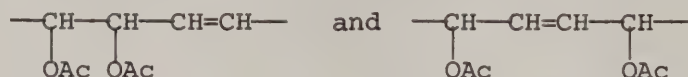
In this review, toxicity refers to those constituents that produce some adverse effect in man or animal, whether it be acutely lethal or merely chronic causing poor growth, hypertrophy of organs or decreased availability of protein, vitamins, and minerals. Many natural constituents of legumes and oilseeds have a deleterious effect on man or animals. Six species of food legumes and six oilseeds are discussed in detail because of their exceptional potential as food-protein sources. The chemical nature of the toxicants, their physiological and biological effects, mechanism of action, and technology required to prepare protein products free of toxic components are emphasized.

### 3775 • Oxidative Acetoxylation of Methyl Oleate with Palladium Catalysts

Edwin N. Frankel, William K. Rohwedder, William E. Neff, and David Weisleder

J. Org. Chem. 40(22): 3247-3253.\* October 1975

Catalytic acetoxylation of methyl oleate in acetic acid produces allylic acetoxy fatty esters in high yield. These derivatives are related to products of autoxidation. Useful catalyst systems include:  $\text{PdCl}_2 + \text{CuCl}_2 + \text{NaOAc}$ ,  $\text{PdCl}_2$  or  $\text{Pd}(\text{OAc})_2 + \text{LiNO}_3$ ,  $\text{Pd}/\text{Al}_2\text{O}_3$  or  $\text{Pd}/\text{C} + \text{LiNO}_3$ , and  $\text{Pd}/\text{C}$ . Esters produced in 50 to 97% conversion at 70-120° and  $\text{O}_2$  at 1 atmospheric pressure or 20-45 p.s.i.g. include acetoxy (20-63%) and diacetoxy (20-47%) octadecenoate. Mono-oxygenated products characterized by gas chromatography-mass spectrometry of the silyl ether derivatives were mainly a mixture of 8-acetoxy-9(*cis/trans*)-, 10-acetoxy-8(*cis/trans*)-, 9-acetoxy-10(*cis/trans*)-, and 11-acetoxy-9(*cis/trans*)-octadecenoate. Di-oxygenated products had both 1,2- and 1,4-diacetoxy allylic structures:



1,3-Disubstituted isomers were ruled out by nuclear magnetic resonance. The  $\text{Pd-LiNO}_3$  catalyst system proved to be highly selective for the allylic acetoxylation of methyl oleate. A mechanism involving allylic  $\text{Pd}(\text{Cl}/\text{OAc})^x$  complex intermediates explains the formation of allylic acetoxy esters. For diacetoxylation a direct pathway from oleate is invoked that involves interconversion of  $\sigma$ -oxypalladation,  $\pi$ -olefin, and  $\pi$ -allyl complex intermediates.

\*Characterization data by IR, GLC, TLC, NMR, MS, and GC-MS appear following these pages in the microfilm edition of this volume. Copies of the supplementary material from this paper are also available from the authors.



## 3776 • Culture Collections and Patent Depositions

T. G. Pridham and C. W. Hesseltine

Adv. Appl. Microbiol. 19: 1-23. 1975

As part of some patent applications, cultures of microorganisms must be deposited in a collection of microorganisms recognized as suitable by the U.S. Patent Office. The practice has continued since the first cultures were deposited in 1948. The ARS Culture Collection is one of the officially recognized depositories; some problems have been encountered in maintaining a special collection of patent deposition strains during the past 24 years. The direction of microbiological and chemical research can affect future activities of patent culture collections.

## 3777 • Antibiotic-Producing Fungi: Current Status of Nomenclature

C. W. Hesseltine and J. J. Ellis

Adv. Appl. Microbiol. 19: 47-57. 1975

This paper reviews the names of fungi known to produce antibiotics and discusses the rules governing the naming of fungi. The points of difference in the rules of nomenclature between the bacterial and the Botanical Code are pointed out. The preparation of descriptions of fungi and the preservation of type cultures are described.

3778 • Unsaturated C<sub>18</sub>  $\alpha$ -Hydroxy Acids in *Salvia nilotica*

M. B. Bohannon and R. Kleiman

Lipids 10(11): 703-706. November 1975

The oil of *Salvia nilotica* Jacq. (Labiatae) seed contains 0.6%  $\alpha$ -hydroxy-oleic, 4.2%  $\alpha$ -hydroxylinoleic, and 5.4%  $\alpha$ -hydroxylinolenic acids. The first two have not been found previously in seed oils. In addition to the common fatty acids, also identified were small amounts of three unsaturated C<sub>17</sub> acids and one branched chain C<sub>17</sub> acid. Methyl esters of the component fatty acids were fractionated by both column and thin-layer chromatography. These esters were identified by combination of gas chromatography, gas chromatography-mass spectrometry, ozonolysis-gas chromatography, infrared, and nuclear magnetic resonance.

## 3779 • Flavor Evaluation of Copper-Nickel Hydrogenated Soybean Oil and Blends with Unhydrogenated Oil

K. J. Moulton, R. E. Beal, K. Warner, and B. K. Boundy

J. Am. Oil Chem. Soc. 52(11): 469-472. November 1975

Soybean oils hydrogenated to zero linolenate in the pilot plant with a mixed copper-nickel catalyst and a straight copper chromite catalyst were

evaluated and compared for flavor and odor. Hydrogenated oils were winterized and deodorized and stabilized with butylated hydroxytoluene, butylated hydroxyanisole, citric acid, and methyl silicone. Taste panel flavor scores of stored oils and room odor scores of oil at frying temperature were similar for oils hydrogenated either with straight copper chromite or with mixed copper chromite-nickel catalysts. Blends containing 1, 2, and 3% linolenate made from unhydrogenated soybean salad oil and soybean oil hydrogenated to 0% linolenate with mixed copper chromite-nickel catalyst were similarly evaluated. Panel responses indicated a blend of 29% unhydrogenated soybean salad oil and 71% hydrogenated soybean oil scored slightly lower than the hydrogenated soybean oil.

3780 • Nylon-9 from Unsaturated Fatty Derivatives: Preparation and Characterization

R. B. Perkins,<sup>1</sup> J. J. Roden, III,<sup>1</sup> and E. H. Pryde

(<sup>1</sup>Southern Research Institute, Birmingham, Alabama)

J. Am. Oil Chem. Soc. 52(11): 473-477. November 1975

Methyl azelaaldehyde, soy nitriles, and oleonitrile were compared as starting materials for making nylon-9. Less difficulty was encountered in purifying intermediates from oleonitrile than from the other two. All three routes involved hydrolysis of methyl 9-aminononanoate to 9-aminononanoic acid. Self-catalyzed hydrolysis of the amino ester in water produced low yields of monomer owing to oligomer formation, but hydrolysis in the presence of barium hydroxide was more successful. Polymerization of the amino acid proceeded smoothly with 9-acetamidononanoic acid used to control molecular weight. Strength and moisture-absorbing properties were determined for nylon-9, as well as for nylon-6/9, -9/11, and -9/12 copolymers.

Nylon-9 has attractive properties, offering tensile yield and flexural strengths approaching those of nylon-6 while having low water absorption (ASTM D-570) approaching closely that of nylon-11 and -12. The cost of producing 10,000,000 pounds per year of nylon-9 from purchased oleonitrile was estimated at \$0.99 per pound at 100% yield and at \$2.18 per pound at 35.5% yield. Further process development is needed before yields in a commercial plant can be forecast.

3781 • Solid Substrate Fermentation of Feedlot Waste Combined with Feed Grains

R. A. Rhodes and W. L. Orton

Trans. ASAE 18(4): 728-733. July-August 1975

A new process is being developed for recycling animal wastes based on a solid-state fermentation with cracked grains. To date, the work has been done mainly with corn and the liquid fraction of cattle feedlot waste.

This liquid, separated by an oscillating 30-mesh screen, contains 10% solids in the form of microbial cells and either fine or soluble waste components; it has a nitrogen content of 3-4 mg./ml. When added to cracked corn at a ratio of 1:2, the thick liquid adheres to grain surfaces. Grain-waste mixtures at about 40% moisture, incubated aerobically by tumbling in a slowly revolving (0.5 r.p.m.) vessel, such as a small cement mixer, rapidly undergo an acid fermentation caused by the selective growth of lactic acid bacteria originally present in the waste; other waste organisms die. The number of bacterial cells increases 100-fold within 24 hours; yeasts emerge after extended incubation. The fetid waste odor quickly disappears as 0.1 meq. of acid per wet gram of fermented solids is generated while nitrogen is conserved. The acids formed are primarily lactic and acetic, with lesser amounts of propionic and butyric.

Exploratory tests with milo and wheat, as well as with wastes from hogs and cattle on different rations, indicate that the new process may be widely applicable. Diluted whole waste may be used when inclusion of fiber in the fermented feed product is of no concern. The solid-state system provides an easily established selective environment for microbial growth. Specific organisms and added inorganic nitrogen may be used for a continuous operation to generate higher protein content in grain-based rations.

- 3782 • Corn Endosperm: Protein Distribution and Amino Acid Composition in Amylomaize vs. Normal Dent Hybrid  
M. J. Wolf, C. C. Harris, and G. L. Donaldson  
Cereal Chem. 52(6): 765-770. November-December 1975

Commercial amylomaize hybrids had a significantly higher proportion of saline-soluble protein and glutelins and a lower ethanol-soluble protein content than normal dent hybrid corn. In agreement with this pattern of protein distribution, lysine content of endosperm protein was significantly higher than that of normal dent hybrid corn, varying from 2.3 g./16 g. N in class 5 amylomaize (62% amylose) to 2.9 g./16 g. N in class 8 amylomaize (81% amylose) as compared with 1.6 g./16 g. N in normal dent hybrid (28% amylose). Other essential amino acids were also higher than those of normal dent hybrid corn but the differences were not significant. In contrast to amylomaize hybrids, endosperm proteins of the high-amylose inbred line W64Aae (61% amylose) were similar to those of the normal counterpart W64A (32% amylose) with respect to both protein distribution and amino acid composition.

- 3783 • Subcellular Distribution and Enzyme Digestibility of Endosperm Proteins of Amylomaize and Normal Corn  
M. J. Wolf and U. Khoo  
Cereal Chem. 52(6): 771-778. November-December 1975



Endosperm storage protein of amylomaize contains protein bodies averaging about 1.16  $\mu\text{m}$ . in diameter as compared with an average of 1.50  $\mu\text{m}$ . for normal dent corn. Both alcohol-soluble and alcohol-insoluble proteins are dispersed throughout the protein bodies of amylomaize. In normal corn, alcohol-insoluble protein is condensed in the central part of the protein body and zein in the peripheral. The protein bodies of amylomaize appear to contain a larger proportion of alcohol-insoluble protein than those of normal dent corn. In addition to protein bodies, numerous smaller structures, about 0.2  $\mu\text{m}$ . in diameter, are embedded in the matrix protein of both amylomaize and normal corn. In both types of corn, proteinase digests both matrix and zein body proteins in thin sections where contents of the bodies are exposed to the enzyme solution. Under these conditions the matrix proteins are more rapidly digested than the proteins of the bodies.

3784 • Corn Proteins: Chemical and Physical Changes During Drying of Grain

J. S. Wall, C. James, and G. L. Donaldson

Cereal Chem. 52(6): 779-790. November-December 1975

Changes in proteins in high-moisture corn dried at elevated temperatures were investigated to determine reasons for previously reported reduced grain quality for seed, milling, and feed uses. Grain harvested at 25% moisture was dried to about 15% moisture by heating at air temperatures ranging from 15° to 143° C. in a forced-air dryer. Proteins were sequentially extracted from defatted grain or endosperm meals with various solvents. Amounts of proteins extracted with 0.5 *N* NaCl were markedly reduced in meals heated to 143° C., and their electrophoretic patterns changed significantly. A smaller decrease occurred in yield of zein extracted with 70% ethanol-0.5% sodium acetate. Buffer containing 0.5% sodium dodecyl sulfate (SDS) dissolved some of the unsolubilized protein. Increased solubility in 0.5% SDS solution containing mercaptoethanol of proteins of corn dried with 143° C. air indicated that intermolecular disulfide bonds are formed during heating. The number of sulfhydryl groups decreased during heating of whole grain; a parallel decrease occurred in grain viability. Lysine and available lysine contents were reduced slightly by heating at the highest temperature.

3785 • Carbohydrate Catabolism of Selected Strains in the Genus *Agrobacterium*

Larry O. Arthur, Lawrence K. Nakamura, Grant St. Julian, and Lee A. Bulla, Jr.

Appl. Microbiol. 30(5): 731-737. November 1975

Radiorespirometric and enzyme analyses were used to reveal the glucose-catabolizing mechanisms functioning in single strains of seven presumed

*Agrobacterium* species. The Entner-Doudoroff and pentose cycle pathways functioned in *A. radiobacter*, *A. tumefaciens*, *A. rubi*, and *A. rhizogenes*. Whereas both catabolic pathways were utilized to an almost equal degree in the *A. radiobacter* and *A. tumefaciens* strains, use of the Entner-Doudoroff pathway predominated in the *A. rubi* and *A. rhizogenes* strains. *A. stellulatum* catabolized glucose almost solely through the Entner-Doudoroff pathway. In *A. pseudotsugae* and *A. gypsophilae*, glucose was metabolized mainly through the Emden-Meyerhof-Parnas pathway; the pentose phosphate pathway was also utilized.

- 3786 • Fibrous Material in Feedlot Waste Fermented by  
*Trichoderma viride*  
 T. Kaneshiro, B. F. Kelson, and J. H. Sloneker  
 Appl. Microbiol. 30(5): 876-878. November 1975

*Trichoderma viride* QM 9123 fermented fiber isolated from feedlot waste at concentrations up to 16.7% solids. The fermented fiber solids decreased by 32%, and carbohydrate decreased by 60%. Cellulolytic enzyme production was better with fiber substrates that had been alkali pretreated and had a lower hemicellulose-to-cellulose ratio.

- 3787 • Isolation of Perloline, the Yellow Alkaloid of Tall Fescue  
 Shelly G. Yates, S. Peter Rogovin, Lowell P. Bush,<sup>1</sup>  
 Robert C. Buckner,<sup>1</sup> and James A. Boling<sup>1</sup>  
 (<sup>1</sup>University of Kentucky and ARS, USDA, Lexington)  
 Ind. Eng. Chem., Prod. Res. Dev. 14(4): 315-319.  
 December 1975

An extraction method developed to isolate perloline for feeding studies in ruminants produced 338 g. of perloline monohydrochloride from 1090 kg. of dehydrated tall fescue forage, which contained 0.14% perloline by thin-layer densitometry analysis. Acid conditions promoted extraction, but interfered with subsequent processing. Perloline losses, due primarily to alkaline degradation and photodecomposition, were minimized by process management. Assays of the free base in chloroform by absorption measurements at 480 nm, but not at 243 nm nor 288 nm, correlated well with thin-layer densitometry. The isolated perloline monohydrochloride showed no antibiotic activity against seven microorganisms, but it was marginally active against P388 lymphocytic leukemia.

3788 • Conversion of 1,4-Anhydroerythritol into 1,4-Anhydro-DL-threitol: Chloride Displacement of Sulfonate

James C. Goodwin and John E. Hodge

Carbohydr. Res. 44(1): 106-111. October 1975

1,4-Anhydroerythritol was converted to DL-1,4-anhydrothreitol in 23% overall yield by chloride displacement of tosyl and 34% overall yield by chloride displacement of mesyl. Methanesulfonation of 1,4-anhydroerythritol with two moles of methanesulfonyl chloride in pyridine at 25° C. for 18 hours produced 2,3-di-O-(methysulfonyl)-1,4-anhydroerythritol as a crystalline compound in 93% yield. The slower reacting *p*-toluenesulfonyl chloride in pyridine at 25° C. for 9 days gave a mixture of 2(3)-hydroxy-3(2)-O-*p*-tolylsulfonyl-1,4-anhydroerythritol and 2,3-di-O-*p*-tolylsulfonyl-1,4-anhydroerythritol, which was resolved into crystalline compounds. 2,3-Di-O-(methysulfonyl)-1,4-anhydroerythritol (1 mole), when refluxed with pyridine hydrochloride (2 moles) in pyridine, produced crystalline 2(3)-chlorodeoxy-3(2)-O-(methysulfonyl)-1,4-anhydrothreitol in 51% yield. Similar treatment of 2,3-di-O-*p*-tolylsulfonyl-1,4-anhydroerythritol gave 2(3)-chlorodeoxy-3(2)-O-*p*-tolylsulfonyl-1,4-anhydrothreitol in 67% yield. By hydrolysis of 2(3)-chlorodeoxy-3(2)-O-(methysulfonyl)-1,4-anhydrothreitol with aqueous sodium hydroxide (5%) for 16 hours at 75° C., DL-1,4-anhydrothreitol was isolated in 71% yield; the yield was lower for 2(3)-chlorodeoxy-3(2)-O-*p*-tolylsulfonyl-1,4-anhydrothreitol (63.4%).

3789 • Starch-Encased Powdered Rubber: Pilot-Plant Production

H. F. Conway and V. E. Sohns

J. Elastomers Plast. 7: 365-371. October 1975

The requirements for increased quantities of elastomer powders for evaluation in direct injection molding on a continuous basis made a larger system for their production necessary. A study of the operations required in the laboratory method suggested that possibly water washing and air drying could be substituted for the more expensive alcohol-dehydration and vacuum-drying steps; subsequently, coagulation on a continuous basis was substituted for the laboratory-scale batch coagulation. In this paper we describe this new process and present a cost estimate for its commercial employment.

3790\* • New Techniques in Lipid Analysis: Isomeric Fatty Acids

H. J. Dutton

In "Analysis of Lipids and Lipoproteins," ed. E. G. Perkins, chap. 9, pp. 138-152. Champaign, Ill. 1975

A review of methods developed at the Northern Regional Research Laboratory for determining the position and geometric configuration of unsaturation, particularly in partially hydrogenated fats.



## 3791\* • Mass Spectrometry of Lipids

William K. Rohwedder

In "Analysis of Lipids and Lipoproteins," ed. E. G. Perkins,  
chap. 11, pp. 170-182. Champaign, Ill. 1975

This short review explains some of the fragmentation patterns that are of greatest concern to the lipid chemist and describes several special techniques that he might find useful.

Many books are now available on mass spectrometry, and there are also excellent reviews of mass spectrometry applicable to lipids. Mass spectra of methyl esters of fatty acids have been thoroughly characterized by Ryhage and Stenhagen and most of the accepted mechanisms for lipid fragmentations are derived from their work.

3792 • Interaction of T-2 Toxin with *Salmonella* Infections of ChickensBoonbungearn Boonchuvit,<sup>1</sup> P. B. Hamilton,<sup>1</sup> and H. R. Burmeister(<sup>1</sup>North Carolina State University, Raleigh)

Poult. Sci. 54(5): 1693-1696. September 1975

A significant ( $P < 0.05$ ) interaction resulting in increased mortality occurred in chickens fed T-2 toxin (16  $\mu\text{g./g.}$  of diet) and infected with either *Salmonella* *worthington*, *S. thompson*, *S. derby*, or *S. typhimurium* var. *copenhagen*, all species that cause paratyphoid. No interaction on growth rate or relative size of the bursa of Fabricius occurred, although T-2 toxin alone caused a significant ( $P < 0.05$ ) regression of that organ. The spleen size relative to the body weight was decreased ( $P < 0.05$ ) by T-2 toxin and increased ( $P < 0.05$ ) by the *Salmonella* infections. Interactions were observed on spleen size between the toxin and *S. thompson* ( $P < 0.05$ ) and *S. derby* ( $P < 0.10$ ). Total serum proteins were not affected by T-2 toxin or *Salmonella* infections. Agglutinins were formed in response to the infections, but the titers were unaltered by T-2 toxin.

## 3793 • Acyl Esters from Oxo-Derived Hydroxymethylstearates as Plasticizers for Polyvinyl Chloride

E. N. Frankel, W. E. Neff, F. L. Thomas, T. H. Khoe,  
E. H. Pryde, and G. R. Riser<sup>1</sup>

(<sup>1</sup>Eastern Regional Research Center, ARS, USDA, Philadelphia, Pa.)  
J. Am. Oil Chem. Soc. 52(12): 498-504. December 1975

Hydroxymethylstearates were made by hydroformylation or oxo reaction of mono- and polyunsaturated fats and esters with either rhodium-triphenylphosphine or cobalt carbonyl catalysts. Rhodium-oxo products were hydrogenated with nickel catalyst, whereas cobalt-oxo products were heated directly under hydrogen pressure. Hydroxymethyl fatty alcohols also were

prepared by a two-step copper-chromite hydrogenation by hydroformylated linseed fatty esters. Of these hydroxymethyl compounds, 39 were converted to their acetates and other acyloxy derivatives and then evaluated as primary plasticizers for polyvinylchloride. For compounds with good compatibility, methyl 9(10)-acetoxymethylstearate and 9(10)-acetoxymethyloctadecyl acetate gave the lowest flex temperature ( $-47^{\circ}\text{C}.$ ). An unusual combination of good compatibility and low flex temperature was obtained with 2-methoxyethyl 9(10)-acetoxymethylstearate. Addition of more than one acetoxymethyl group in the fatty acid molecule, made possible by rhodium hydroformylation, imparted good compatibility and outstanding permanence (low migration and volatility) but raised flex temperature. Butyl diacetoxymethylstearate, methyl triacetoxymethylstearate, and polyacetoxymethyloctadecyl acetate from linseed esters displayed good compatibility, strength, and volatility characteristics. As glycerides, acetoxymethylated safflower and linseed oils produced good compatibility and outstanding permanence, better than esters commonly used as commercial plasticizers.

3794\* • Aflatoxin Production and Lepidopteran Insect Injury on Corn in Georgia

N. W. Widstrom,<sup>1</sup> A. N. Sparks,<sup>1</sup> E. B. Lillehoj, and W. F. Kwolek<sup>2</sup>

(<sup>1</sup>Southern Grain Insects Research Laboratory, Tifton, Georgia;

<sup>2</sup>Biometrician, North Central Region, ARS, USDA, Peoria, Illinois)  
J. Econ. Entomol. 68(6): 855-856. December 1975

Bagging ears of a dent corn hybrid in the field was effective in reducing damage by naturally occurring populations of Lepidoptera, but was not effective in reducing the production of aflatoxin on the ear. Insects produced conditions in the infested ear that were conducive to aflatoxin production and insect species differed in their relationship to increased aflatoxin production. Ears infested with European corn borer, *Ostrinia nubilalis* (Hubner), had the highest concentrations of aflatoxin B<sub>1</sub>.

3795 • Separation of Spores and Parasporal Crystals of *Bacillus thuringiensis* in Gradients of Certain X-Ray Contrasting Agents

Eugene S. Sharpe, Kenneth W. Nickerson, Lee A. Bulla, Jr., and John N. Aronson<sup>1</sup>

(<sup>1</sup>State University of New York at Albany, Albany, New York)

Appl. Microbiol. 30(6): 1052-1053. December 1975

Spores and parasporal crystals of *Bacillus thuringiensis* can be separated at moderate centrifugation speeds (10,000 to 12,000 r.p.m.) in gradients of Renografin<sup>®</sup> or sodium diatrizoate.

- 3796 • Complexes of Carbohydrates with Aluminate Ion. Chromatography of Carbohydrates on Columns of Anion-Exchange Resin (Aluminate Form)  
Jacob A. Rendleman, Jr. and John E. Hodge  
Carbohydr. Res. 44(2): 155-167. November 1975

With water as sole eluant, the retention volumes for carbohydrates on an aluminate-resin column generally decrease in the order ketoses>aldoses>alditols>methyl glycosides; retention increases with molecular size. Both aluminate ion and hydroxide ion contribute to the chromatographic properties of an aluminate resin. To avoid alkaline degradation and interconversion, which can occur extensively at 25° C., chromatography of reducing sugars must be performed both rapidly and at low temperature. A mixture of D-glucose and D-fructose can be completely separated on a short aluminate column.

- 3797 • Gas-Liquid Chromatography-Mass Spectrometry of Methylated and Deuteriomethylated Per-O-Acetyl-Aldononitriles from D-Mannose  
Fred R. Seymour, Ronald D. Plattner, and Morey E. Slodki  
Carbohydr. Res. 44(2): 181-198. November 1975

Peracetylated aldononitriles of the tetra-, tri-, and di-methyl ethers of D-mannopyranose were separated by gas-liquid chromatography, and analyzed by mass spectrometry. Through introduction of deuteriomethyl ether groups, various fragment ions constituting the mass spectra were identified and related to the parent methylated sugar structures. Also identified were several characteristic series of fragment ions that are common to two or more methylated D-mannopyranosides. As expected, mass spectra of the D-mannose derivatives were identical to those previously observed for D-glucose methylated in the same positions. Distinctive mass spectra were also recorded for all additional di-O-methyl-D-mannose derivatives. This information permits use of peracetylated aldononitrile derivatives in methylation-fragmentation analysis of aldohexans.

- 3798 • Lipid Metabolism During Bacterial Growth, Sporulation, and Germination: Differential Synthesis of Individual Branched- and Normal-Chain Fatty Acids During Spore Germination and Outgrowth of *Bacillus thuringiensis*  
Kenneth W. Nickerson, Lee A. Bulla, Jr.,<sup>1</sup> and Timothy L. Mounts  
(<sup>1</sup>U.S. Grain Marketing Research Center, ARS, USDA,  
Manhattan, Kansas)  
J. Bacteriol. 124(3): 1256-1262. December 1975

The biosynthesis of individual branched- and normal-chain fatty acids during *Bacillus thuringiensis* spore germination and outgrowth was studied by comparing pulsed and continuous labeling of these fatty acids with



[U-<sup>14</sup>C]acetate. The relative specific activity of each fatty acid varies with time as the cell progresses through outgrowth. However, fatty acid synthesis does occur in two distinct phases. Upon germination, acetate is incorporated only into the iso-isomers i-C<sub>13</sub>, i-C<sub>14</sub>, and i-C<sub>16</sub>; no normal or anteiso synthesis occurs. Subsequent to T<sub>30</sub>, the full complement of branched- and normal-chain homologues is formed and there is a dramatic enhancement in the overall rate of fatty acid synthesis. Significantly, this rate increase coincides with a marked shift from the synthesis of short-chain to long-chain fatty acids. These findings illustrate a dichotomy in synthesis that may result from initial fatty acid formation by preexisting spore fatty acid biosynthetic enzymes in the absence of de novo protein synthesis. Elucidation of the timing and kinetics of individual fatty acid formation provides a biochemical profile of activities directly related to membrane differentiation and cellular development.

3799 • Profiles for pH, Temperature, and Dissolved O<sub>2</sub> Levels in Enzyme Production: Monitoring in Small-Scale Fermentors

R. E. Peterson and G. E. N. Nelson

Biotechnol. Bioeng. 17(12): 1839-1845. December 1975

A 1-liter table-top fermentor was monitored for pH, temperature, and dissolved O<sub>2</sub> level during growth studies of *Erwinia aroideae*, *Pseudomonas testosteroni*, and *Proteus vulgaris*. The respective profiles were then related to L-asparaginase (E.C. 3.5.1.1), L-glutaminase (E.C. 3.5.1.2), and L-serine dehydratase (E.C. 4.2.1.13) production. By using multispan, multizero range recorders as scale expansion units, inflections were observed during fermentations that graphically illustrate metabolic changes. Significant pH fluctuations signal the various phases of microbial growth. Feedback of pH is suggested to control nutrient flow in continuous fermentations to optimize product formation.

3800 • Treating Shelled Corn with Ammonia--Application Via Ammonia-Air Mixtures

E. B. Lancaster, A. C. Stringfellow, and O. L. Brekke

Trans. ASAE 18(6): 1158-1161, 1164. November-December 1975

Small-scale laboratory experiments were undertaken to develop a method for gas-phase ammoniation of corn. The method offers a potential, low-cost means for increasing nonprotein nitrogen content of the grain, and for treating shelled high-moisture corn with ammonia to modify typical microbiological activity during storage. Ammonia mixed with air was recycled through a small glass column (2.7 inches in diameter) holding in a bed 37-48 inches deep 5 pounds of corn containing either 13, 17, or 26% moisture content. From 1/2 to 4% ammonia, based on dry matter content of the corn, was added during 1 to 3 hours; after 24 hours of gas recirculation,

the ammonia was uniformly distributed throughout the bed. Temperature of the corn and composition of the gas leaving the column during treatment were recorded.

Partition of ammonia between the gas and solid at equilibrium can be approximated from a simple absorption theory. However, heat and mass transport effects before equilibrium are quite complex. Suggestions are made on how data can be handled in a computer simulation of the entire system.

3801\* • Development and Characteristics of a Disomic-1D Addition Line of Durum Wheat

L. R. Joppa,<sup>1</sup> J. A. Bietz, and C. McDonald<sup>2</sup>

(<sup>1</sup>Agric. Res. Serv., USDA, Fargo, North Dakota; <sup>2</sup>North Dakota State University, Fargo)

Can. J. Genet. Cytol. 17: 355-363. 1975

A pair of chromosomes from Chinese Spring hexaploid wheat (*Triticum aestivum* L.) was added to the tetraploid durum wheat (*Triticum turgidum* L.) cultivar Langdon. Crosses between this disomic-addition line and Chinese Spring ditelosomic 1DL produced progeny with 14 pairs + 1 heteromorphic bivalent + 6 univalents (14" + 1" + 6'). Therefore, this durum line with 15 pairs of chromosomes is a 1D-disomic addition line to Langdon durum wheat. Seed from the 1D-disomic addition line produced two high-molecular-weight glutenin polypeptide subunits not present in durum wheats, as determined by sodium dodecyl sulfate-polyacrylamide gel electrophoresis. These polypeptides were similar to those present in Chinese Spring. Semolina milled from seed of the 1D-disomic addition line had very strong mixing properties, as determined by the micro-mixograph, and 2% higher protein than the recurrent parent Langdon or other durum varieties. The amino acid content of the line was similar to that of the other durums.

3802 • Kenaf Roots--A Pulping Resource

M. O. Bagby, R. L. Cunningham, and W. C. Adamson<sup>1</sup>

(<sup>1</sup>U.S. Plant Introduction Station, Savannah, Georgia)

Tappi CA Report No. 58, "Non-Wood Plant Fiber Pulping--Progress Report No. 6," pp. 63-68. Atlanta, Ga. October 1975

Chemical and physical properties were determined of root stubble and of roots from kenaf plants harvested green and after frost by 7 weeks and 3 months, as well as their soda pulps. Collecting roots could increase the yield of useful fiber by at least 10%. With properties generally intermediate to those of core and whole stem, characteristics of both uncooked and cooked roots suggest that they are core-rich. Screened pulp yield based on cleaned root fiber ranged from 45.1% for aged stubble to

52.7% for 7 weeks post-frost roots with respective Kappa numbers of 25.5 and 30.8. These data can be contrasted with stem-screened pulp yield of 50.0 to 53.4% with Kappa numbers of 18.6 and 15.9

3803 • Kenaf Stem Yield and Composition: Influence of Maturity and Field Storage

M. O. Bagby, W. C. Adamson,<sup>1</sup> T. F. Clark, and G. A. White<sup>2</sup>  
(<sup>1</sup>U.S. Plant Introduction Station, Savannah, Georgia; <sup>2</sup>Germplasm Resources Laboratory, Beltsville, Maryland)  
Tappi CA Report No. 58, "Non-Wood Plant Fiber Pulping--Progress Report No. 6," pp. 69-72. Atlanta, Ga. October 1975

As an annual crop for papermaking fibers, kenaf can be harvested either green or after a killing frost. Extended harvest schedules, planting density, and location all influence yield and chemical composition. From planting densities of 100,000 and 200,000 plants per hectare at Glenn Dale, Maryland, maximum yields of dry matter at frost were 11.6 and 12.2 metric tons per hectare, respectively. In an identical sequence at Fleming, Georgia, the maximum yields, 15.7 and 15.8 metric tons per hectare, occurred one month after frost. In both locations extractable substances decreased after frost; whereas relative percentages of cellulose and pentosan increased. Even 3 months after frost, cellulosic content remained at least equal to that 6 weeks before frost.

3804 • Economic Potential of Kenaf Production

Clarence A. Moore,<sup>1</sup> Warren K. Trotter,<sup>2</sup> Ray S. Corkern,<sup>3</sup> and Marvin O. Bagby  
(<sup>1</sup>Econ. Res. Serv., USDA, Peoria, Illinois; <sup>2</sup>Econ. Res. Serv., USDA, New Orleans, Louisiana; <sup>3</sup>Econ. Res. Serv., USDA, Athens, Georgia)  
Tappi CA Report No. 58, "Non-Wood Plant Fiber Pulping--Progress Report No. 6," pp. 73-80. Atlanta, Ga. October 1975

The economic potential of kenaf resides ultimately with the pulp and paper industry management. They will decide when the tree supply is becoming sufficiently scarce and kenaf's cost and capability sufficiently strong to bring it on stream. Company land capable of growing kenaf would, at present, provide dry material at roughly half the cost of producing pulpwood. Three to five times as much per hectare could be produced annually. If priced at 1974 delivered prices for pulpwood, kenaf would compete well with the major crops in the South at yields from 10 metric tons to 15 metric tons per hectare.



- 3805\* • The Search for New Sweeteners  
 George E. Inglett  
*In* "Sweeteners--Issues and Uncertainties," Proc. 4th Acad.  
 Forum, March 25-26, 1975, pp. 207-211. National Academy of  
 Sciences, Washington, D.C. 1975

In the formulation of food products, a variety of nutritive as well as nonnutritive sweeteners afford the best opportunities for providing consumers with excellent foods. In order to have successful food products, there are certain sensory and functional properties that sweeteners must fulfill. A variety of sweeteners is the best approach to this problem. Some new sweeteners are considered as potential alternatives.

#### REPUBLICATIONS

- 2666\* • Composicion de la Planta y del Grano de Sorgo  
 [Composition of Sorghum Plant and Grain, chap. 4, pp. 118-166]  
 Joseph J. Wall and Charles W. Blessin  
*In* "Produccion y Usos del Sorgo," eds. Joseph S. Wall and  
 William M. Ross, chap. 4, pp. 69-92. 1975
- 2667\* • Consideraciones de Orden Economico [Economic Considerations,  
 chap. 18, pp. 627-664]  
 Clarence A. Moore<sup>1</sup> and Kenneth R. Majors<sup>2</sup>  
 (<sup>1</sup>Econ. Res. Serv., USDA, Peoria, Illinois; <sup>2</sup>Fed. Ext. Serv.,  
 USDA, Peoria, Illinois)  
*In* "Produccion y Usos del Sorgo," eds. Joseph S. Wall and  
 William M. Ross, chap. 18, pp. 355-375. 1975
- 2668\* • El Futuro del Sorgo Granifero [The Future of Grain Sorghum,  
 chap. 19, pp. 665-686]  
 Clarence A. Moore<sup>1</sup> and D. G. Nelson<sup>2</sup>  
 (<sup>1</sup>Econ. Res. Serv., USDA, Peoria, Illinois; <sup>2</sup>Grain Sorghum  
 Producers Association, Amarillo, Texas)  
*In* "Produccion y Usos del Sorgo," eds. Joseph S. Wall and  
 William M. Ross, chap. 19, pp. 377-389. 1975

The book "Sorghum Production and Utilization," edited by Joseph S. Wall and William M. Ross, was published in 1970. This work has been translated into Spanish, English titles and original page numbers are given in brackets for these three chapters.

- 3240 • Mycotoxins. Aflatoxins: Detection and Determination  
[A. Ciegler]  
North. Reg. Res. Lab., U.S. Agric. Res. Serv., CA-NRRL-37(Rev.),  
2 pp. November 1975 [Processed]

This is a revised version of the Correspondence Aid originally issued in December 1972.

#### UNOFFICIAL PUBLICATION

Listing of publications and patents of the Northern Regional Research Laboratory would not be complete without including some unofficial publications. These are writings by members of the Northern Laboratory staff, and, although written from previously published official material, are of a public service value from the standpoint of review and updating of the literature. Reprints are not available at the Northern Regional Research Laboratory for distribution.

#### Bacterial Citrate Synthases: Purification, Molecular Weight and Kinetic Mechanism

Donovan E. Johnson and Richard S. Hanson<sup>1</sup>

(<sup>1</sup>University of Wisconsin, Madison)

Biochim. Biophys. Acta 350(2): 336-364. June 1974

## CONTRACT AND GRANT RESEARCH PUBLICATIONS

[Report of research done by an outside agency under contract with the U.S. Department of Agriculture and supervised by the Northern Regional Research Laboratory.]

- 268-C\* • Rapid and Simple Method for the Determination of Tryptophan in Cereal Grains  
Jose Madrid Concon  
University of Kentucky, Lexington  
Anal. Biochem. 67(1): 206-219. July 1975
- 271-C\* • Chemical Determination of Critical Amino Acids in Cereal Grains and Other Foodstuffs  
Jose Madrid Concon  
University of Kentucky, Lexington  
*In* "Protein Nutritional Quality of Foods and Feeds. Part I. Assay Methods--Biological, Biochemical, and Chemical," ed. Mendel Friedman, chap. 18, pp. 311-379. 1975
- 273-C\* • Interference of Ethanol in the Determination of Tryptophan by the Method of Spies and Chambers  
Jose Madrid Concon  
University of Kentucky, Lexington  
Anal. Biochem. 68(2): 643-647. October 1975

[Report of research done by an outside agency under a grant from the U.S. Department of Agriculture and supervised by the Northern Regional Research Laboratory.]

- 142-G\* • The Structure of V Amylose Dehydrate: A Combined X-Ray and Stereochemical Approach  
Vincent Gerard Murphy,<sup>1</sup> Bert Zaslow,<sup>2</sup> and Alfred Dexter French<sup>1,2</sup>  
(<sup>1</sup>Southern Regional Research Laboratory, New Orleans, Louisiana;  
<sup>2</sup>Arizona State University, Tempe)  
Biopolymers 14(7): 1487-1501. July 1975



[Report of research work supported with funds provided by the U.S. Department of Agriculture under the authority of U.S. Public Law 480, 83rd Congress, and sponsored by the Northern Regional Research Laboratory.]

- 363-F • Pyrethrins from *In Vivo* & *In Vitro* Tissue Culture of *Tagetes erecta* Linn.  
Pushpa Khanna, Raka Sharma, and Renu Khanna  
University of Rajasthan, Jaipur, India  
Indian J. Exp. Biol. 13(5): 508-509. September 1975

July-December 1975

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## PATENTS

[These patents are assigned to the Secretary of Agriculture. Copies of patents may be purchased (50 cents each) from the Commissioner of Patents and Trademarks, U.S. Patent and Trademark Office, Washington, D.C. 20231. Order by number, do not send stamps.]

### Recovery and Reactivation of Rhodium Hydroformylation Catalysts

John P. Friedrich

U.S. Patent 3,899,442. August 12, 1975

Soluble rhodium materials are concentrated in the residue from the distillation of hydroformylated products resulting from the reaction of unsaturated fatty compounds, unsaturated fatty acids, or unsaturated fatty compounds with carbon monoxide and hydrogen in the presence of a supported rhodium catalyst. An active catalyst was provided when the rhodium was recovered and resupported. The same rhodium was recovered and resupported 10 times with no significant loss in activity.

### Thiolation of Polysaccharides

Donald Trimmell, Baruch S. Shasha, and William M. Doane

U.S. Patent 3,914,214. October 21, 1975

A method is disclosed which provides for the replacement of polysaccharide -OH groups with -SH groups by pyrolytic degradation of dithiobis(thioformate) polysaccharide derivatives followed by saponification of the resulting dithiocarbonate ester.

### Feed Additive for Poultry from Soybean Oil Soapstocks

Robert E. Beal

U.S. Patent 3,916,031. October 28, 1975

A poultry feed additive is described which equals the feed efficiency of commercial feed fats and which gives significantly better shank pigmentation. The process by which it is obtained has the added advantage of eliminating the pollution factor of a vegetable oil refining effluent.

### Flame-Retardant Polyurethane Foams

Felix H. Otey, Richard P. Westhoff, and Charles L. Mehlretter  
U.S. Patent 3,926,868. December 16, 1975

Highly flame-resistant rigid foams are obtained by the use of novel halogen-containing polyols in the polyurethane synthesis.

### Selective Hydrocarboxylation of Unsaturated Fatty Compounds

Edwin N. Frankel  
U.S. 3,928,231. December 23, 1975

An improved process is described for preparing carboxy acid products in high yields by reacting without isomerization unsaturated vegetable oil material with carbon monoxide and water in the presence of a highly selective catalyst system.

## LICENSING OF PATENTS

Many inventions and discoveries of the Northern Laboratory are covered by patents assigned to the Secretary of Agriculture.

Assigned patents are available for use by business and industry under either exclusive or non-exclusive licenses. Conditions

applicable to the granting of licenses are set forth in the Federal Register, May 14, 1970 [35(94): 7493-7495]. Further information can be obtained from the Administrator, Agricultural Research Service, U.S. Department of Agriculture, Washington, D.C. 20250.



The Northern Regional Research Laboratory is part of the Agricultural Research Service of the U.S. Department of Agriculture. Congress in 1938 authorized four regional laboratories to conduct broad and complex investigations in the field of chemistry and related physical sciences to expand and improve the marketability of agricultural commodities. The addresses and commodities covered are:

<u>Laboratory</u>	<u>Principal Fields of Research</u>
Eastern Regional Research Laboratory 600 East Mermaid Lane Philadelphia, Pennsylvania 19118	Animal products: Dairy, meats, fats, and leather. Plant products: Eastern fruits and vegetables, along with honey.
Northern Regional Research Laboratory 1815 North University Street Peoria, Illinois 61604	Corn, wheat, grain sorghum, oats, soybeans, flax, crambe, and new crops,
Southern Regional Research Laboratory P.O. Box 19687 New Orleans, Louisiana 70179	Cotton and cottonseed; sweet potatoes; rice; grain sorghum, peanuts, and cane sugar.
Western Regional Research Laboratory Berkeley, California 94710	Western fruits, tree nuts, and vegetables; poultry products; forage crops; wheat, barley, rice; wool and mohair; dry beans and peas; castor beans; and safflower.







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